Ni base nanoparticles (NPs) are characterized under low dose conditions in TEM mode. These nanoparticles are mainly designed to act as catalysts in energy devices. Ni, NiO and Pt@NiO nanoparticles are investigated. Particularly the use of NiO@Pt for solar cells (artificial photosynthesis) is attractive, namely the hydrogen evolution center, while NiO has been tested as a catalyst for the oxygen evolution center. Consequently an atomic characterization of the involved nanocrystals is of particular importance. Here, transmission electron microscopy is used with the objective to determine nature, shape and atomic distribution of Pt for different loadings (0-16 at.%) on a Ni core basis. In all cases the electron dose rate has been kept in the range 20-150 e/Å²s in order to avoid surface rearrangement by interaction with the electron beam. The TEAM 05 (80 KeV) has been used together with focal series reconstruction (EWR) to recover both phase and amplitude images that provide information of the spacing and the chemical nature of the corresponding atomic columns. Two procedures have been used for synthesis of nanoparticles. One of them produces Ni and the other NiO-NPs. NiO NPs are then covered with different loadings of Pt in order to create incomplete core shell structures but with superior catalytic activity. Figure 1 shows phase images of Ni NPs, their size varies from 1 to 7 nm and can agglomerate most likely due to their magnetic characteristics. The dose rate used to acquire the experimental images is 30 e-/Å²s. Figure 1b shows experimental images of NiO NPs acquired with a dose rate of 120 e-/Å²s, their average size is around 1.5 nm. During processing Pt is deposited on NiO particles and a typical example is given in the phase images shown in Figs. 2a-b, the dose rate is around 55 e-/Å²s and the Pt coverage is nominally 8 at. %. The nanoparticles have mostly irregular shapes. There is a negligible particle transformation due to the weak interaction with the electron beam. These NPs are nevertheless susceptible to alteration in shape and structure as a consequence of electron beam sample interaction. An example is given in the phase images shown Figs. 3 a-c. In these cases, the dose rate has been increased from 55 e-/Å²s (Fig. 3a) to 300 e-/Å²s (Fig 3b) and 1400 e-/Å²s (Fig. 3c). The particle under observation initially losses atoms that apparently redeposit on the carbon support and migrate (partially) to form a new crystal. The selected NP becomes bicrystalline at the end of this experiment that clearly shows the need to use a proper electron dosage for observation and the possible large influence of thermal effects. Phase images have been used for simulation in order to determine the Pt coverage.

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Fig. 1: Figure 1. (a) Phase image of Ni nanoparticles and (b) Experimental image of NiO nanoparticles at a dose rate of 150 e-/Å²/s.

Fig. 2: Fig. 2. Phase images of NiO nanoparticles with a Pt coverage of 8 at.% and taken with a dose rate of 55 e-/Å²s.

Fig. 3: Fig. 3. Phase images of NiO nanoparticles with an 8 at.% Pt coverage. (a) Dose rate of 55 e-/Å²/s. (b) Dose rate of 300 e-/Å²/s and (e) Dose rate of 1400 e-/Å²/s.